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(54) PRODUCTION OF SYNTHETIC SILICA POWDER

(57)Abstract:

PURPOSE: To provide a process for producing synthetic silica powder having high bulk density and useful for the production of synthetic quartz glass free from void defect.

CONSTITUTION: The objective synthetic silica powder can be produced by hydrolyzing methyl silicate with ammonia water, subjecting the hydrolyzate to polycondensation to obtain colloidal silica, sieving the product to collect particles of $\leq 500\mu\text{m}$ diameter, heat-treating in an oxidizing atmosphere at 1000-1100°C and densifying by introducing helium gas into the reaction system while keeping the above heat-treatment temperature.

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TITLE: Silica powders prodn. - involves hydrolysing
methy: silicate with ammonia water,
polycondensing
forming colloidal silica which is classified
and heating
in oxidative atmos.

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ABSTRACTED-PUB-NO: JP 05017122A

BASIC-ABSTRACT:

Prodn. involves 1) hydrolysing methylsilicate with ammonium hydroxide and is polycondensated to form colloidal silica; 2) classifying the silica into particles up to 500 microns dia; 3) heating at 1000-1100 deg. C in an oxidative atmos; then changing the atmos. to He gas atmos., so that dense silica particles are produced.

USE/ADVANTAGE - Silica powders with high bulk density can be obtd. and are used to produce quartz glass free of foams.

In an example, methylsilicate and 20.5% ammonium hydroxide were dropped simultaneously at a rate of 25.8 litre/hr to obtain silica flocs. They were sieved with a mesh 500 microns and dehydrated with a filter cloth and washed

with water five times. The resulting colloidal silica was put in a quartz glass tube and heated to 1000 deg. C in O₂ gas atmos. for 2 hrs, then in He gas atmos. for 3 hours to obtain silica powders with 0.98 bulk density

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 CLAIMS

[Claim(s)]

[Claim 1] 500 after hydrolyzing, carrying out the polycondensation of the methyl silicate and making it into colloidal silica with aqueous ammonia. The manufacture approach of the synthetic silica powder characterized by making it below a screen exception, heat-treating by 1,000 - 1,100 ** under an oxidizing atmosphere, and subsequently introducing and carrying out ebullition of the gaseous helium in this temperature field.

[Translation done.]

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] The manufacture approach of synthetic silica powder, especially bulk specific gravity of this invention are high, and it relates to the manufacture approach of the synthetic silica raw material powder for manufacturing synthetic quartz glass without a poor bubble.

[0002]

[Description of the Prior Art] Although it has the place known well that synthetic quartz glass is obtained according to melting vitrification of synthetic silica powder, about the manufacture approach of this synthetic quartz glass, the Verneuil's method, the soot method, the sol-gel method, the cristobalite-vacuum fusion method, etc. are known.

[0003] Although this Verneuil's method carries out acid hydrogen hydrolysis fusion of the silicon tetrachloride Since an OH radical content is set to about 1,000 ppm at this, there is disadvantage that elevated-temperature viscosity is low. Although the soot method hydrolyzes a silicon tetrachloride in a low-temperature acid hydrogen flame comparatively, and makes a porous glass base material, heating fusion is carried out and transparence vitrification is carried out under a gaseous helium ambient atmosphere, a helium-chlorine gas ambient atmosphere, or a vacuum ambient atmosphere Also for this, the amount of OH radicals or Cl content is 300 ppm. There is a trouble of elevated-temperature viscosity being low since it becomes extent, and deforming at an elevated temperature.

[0004] Moreover, carry out heating fusion of the gelation desiccation object which this sol-gel method hydrolyzed silicate, made the silica sol, and gelled and obtained this with 1,200 ** extent, and although transparence vitrification is carried out An OH radical tends to remain in this, the high thing of elevated-temperature viscosity is hard to be obtained, and manufacture takes long duration. There is disadvantage that only sheet metal is made since generating of a crack may furthermore break out during manufacture. Although this cristobalite-vacuum fusion method solates fumed silica in an alkali water solution, and calcinates and cristobalite-izes it after freeze drying and vacuum fusion is carried out above 1,700 ** There is a fault that the quartz glass of purity obtained by this is bad, and becomes that in which this has a detailed bubble.

[0005]

[Problem(s) to be Solved by the Invention] Therefore, this invention persons examine the approach of manufacturing the high synthetic quartz glass of elevated-temperature viscosity with this sol-gel method. Especially about this, use methyl silicate as silicate, and this is hydrolyzed with aqueous ammonia. Carry out a polycondensation and heat the obtained silica, and after carrying out oxidation clearance of the unreacted organic substance, heat more than 1,500 ** and it is made to sinter in a vacuum. If heating fusion is carried out above 1,700 **, the high synthetic quartz glass of elevated-temperature viscosity subsequently, easily The umbrella consistency of the synthetic silica powder used for this here is low. and the thing which can be acquired cheaply -- having found out (referring to JP,2-1870723,A) -- It turned out [that the synthetic quartz glass obtained as imperforation is inadequate becomes that in which purity is bad and contains a micro bubble / disadvantageous] that it is.

[0006]

[Means for Solving the Problem] It is 500 micrometers after this carrying out the hydrolysis polycondensation of the methyl silicate with aqueous ammonia about the manufacture approach of the synthetic silica powder for synthetic quartz glass manufacture that this invention solved such disadvantage and considering as colloidal silica. It is made below a screen exception, and it heat-treats at 1,000-1,100 degrees C under an oxidizing

atmosphere, and is characterized by subsequently introducing and carrying out eburation of the gaseous helium in this temperature field.

[0007] Namely, the result of having examined many things about the manufacture approach of the synthetic silica powder by which this invention persons had high bulk specific gravity, and eburation was carried out, The polycondensation of the methyl silicate is hydrolyzed and carried out with aqueous ammonia, and it is colloidal silica 500 mum It is made below a screen exception with wet. If gaseous helium is introduced into this temperature field after heat-treating by 1,000 - 1,100 ** under an after [dehydration and washing] oxidizing atmosphere It checked that the synthetic quartz glass which this silica powder has high bulk specific gravity, heating fusion of a header and the silica powder which carried out in this way and was obtained is carried out for becoming that by which eburation was carried out, and synthetic quartz glass, then whose purity are high, and has neither a bubble nor a foreign matter could be obtained easily, and this invention was completed. This is explained further in full detail below.

[0008]

[Function] This is colloidal silica obtained by hydrolysis with methyl silicate and aqueous ammonia, and the polycondensation about the manufacture approach of synthetic silica powder for this invention to manufacture the synthetic quartz glass of a perfect non-bubble by the super-high grade 500 mum It is made below a screen exception with wet, it calcinates by the oxidizing atmosphere after dehydration and washing, and let it be a summary to introduce and come to carry out eburation of the gaseous helium by 1,000 - 1,100 ** after carrying out degasifying, dehydration and.

[0009] Although manufacture of the synthetic silica powder by this invention is performed with a sol-gel method, the silicate used here is used as methyl silicate, and hydrolysis of this methyl silicate is performed in existence of ammonia. If the polycondensation of this methyl silicate is hydrolyzed and carried out with aqueous ammonia the silica generated by this hydrolysis -- grain size 500-1,000nm It becomes the primary particle of a silica. it is -- This primary particle is spherical, and a ***** OH radical is not contained inside, but an OH radical comes to exist only in a front face, and Even if a puncturing pore diameter is large and applies temperature, it does not hole-closing-ize, but this particle is a surface OH radical and internal C. With heating, it is easily removable, and further, this Motoshige Mitsugi condensation is performed regularly and structure serves as dense colloidal silica.

[0010] Thus, although it is necessary to remove this according to a screen since it is difficult for this gel silica for hole-closing-ization to carry out dehydration and degasifying quickly, therefore thoroughly, although the made colloidal silica adheres to the wall and impeller of a reaction flask, it exfoliates and it becomes a hard gel silica therefore, at this invention, this is 500 according to levigation. mum It considers as the following.

[0011] Thus, although the silica carried out the screen exception is that from which the organic substance contained with heating by the oxidizing atmosphere here is removed (decarbonized), carrying out solid liquid separation of this thing with a conventional method in advance of that processing should just often perform this with a centrifugal dehydrator. If ammonia remains in this centrifugal hydroextraction, it is C by the next oxidation heating. Since it becomes impossible to remove enough, it is required to remove ammonia and a methanol well with ultrapure water. Although it is necessary to perform this by 1 and 00 - 1,100 ** since the opening of the particle comrade of floc is closed and it may become a bubble, if it becomes inadequate [1,000 ** Suemitsu] decarbonizing it and it makes this heat-treatment temperature higher than 1,100 **, although the colloidal silica obtained by such solid liquid separation is subsequently decarbonized with heating by the oxidizing atmosphere, according to this, dehydration is also performed with decarbonization.

[0012] Although the silica powder which carried out this invention in this way, and was decarbonized is contacted to inert gas in this processing temperature It is good, and according to this, eburation of the floc of silica powder is carried out, it becomes the thing of the almost same consistency as glass, and this inert gas raises gaseous helium, then the pack density at the time of synthetic quartz glass manufacture, Moreover, since a charge can be made [more] than before, the synthetic quartz glass which was excellent in quality can be manufactured with sufficient productivity, and the profitableness that there shall not be a bubble and a foreign matter about the synthetic quartz glass obtained is given. Although it is to carry out thermal diffusion to homogeneity, and for one-piece floc [one piece] eburation to have priority over a floc comrade's sintering, and to happen when this reason introduces gaseous helium, such effectiveness is not expectable by any gas other than helium.

[0013]

[Example] Next, the example and the example of a comparison of this invention are given.

Since condensation colloidal silica was obtained in o'clock in 10kg /when simultaneous dropping of methyl silicate 26.5/o'clock, and 20.5% of the aqueous ammonia and EL grade [Omori chemically-modified Manufacture Trade name] 26.5 l./o'clock was carried out at the 25.8l. [/o'clock] dropping rate at the continuation flask of an example and 5l. of examples of a comparison, it is this 500 mum It carried out the screen exception with the network. Subsequently, once it left this thing, solid liquid separation was carried out with the centrifugal dehydrator which formed the filter cloth of 1,000made from polypropylene #, ultrapure water washed 5 times, and the obtained condensation colloidal silica was put in the quartz-glass reactor core tube, and temperature up was carried out over 10 hours from a room temperature to 1,000 ** under the oxygen gas ambient atmosphere, and it held for 2 hours and was decarbonized.

[0014] Although the silica powder which carried out eburnation was obtained when this ambient atmosphere was changed with the gaseous helium ambient atmosphere and held for 3 hours, holding this temperature next They are these fine particles 150-200 The place compared with what did not perform hole-closing-ized processing under this gaseous helium ambient atmosphere for the comparison of this while considering as a mesh, As opposed to the thing of this invention being 0.98 in the bulk-specific-gravity comparison of a tapping method The thing of the example of a comparison was 0.80, when vacuum heating of this thing was carried out after neglect for two days and that amount of degasifying was investigated, to the thing of this invention being A curve as shown in drawing 1 , the thing of the example of a comparison is as B curve, and it was checked that this invention article is that to which it is quite hard to stick.

[0015]

[Effect of the Invention] About the manufacture approach of synthetic silica powder, this invention hydrolyzes methyl silicate with aqueous ammonia, as this was described above. 500 after carrying out a polycondensation and considering as colloidal silica mum Although characterized by making it below a screen exception, heat-treating to an oxidizing atmosphere by 1,000 - 1,100 **, and subsequently introducing and carrying out eburnation of the gaseous helium to it in this temperature field Since the silica powder obtained with the sol-gel method becomes that by which bulk specific gravity was high and was made nonporous according to this Since this can also make [many] raising pack density and a charge when considering as synthetic quartz raw materials for glass the synthetic quartz glass which was excellent also in quality can improve [manufacture cone *****] productivity, synthetic quartz GARASU ** obtained will be obtained if there shall be no poor bubble, and profitableness is given.

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(54)【発明の名称】 合成シリカ粉の製造方法

(57)【要約】 (修正有)

【目的】 本発明はカサ比重が高く、泡不良のない合成石英ガラスを製造するための合成シリカ粉の製造方法の提供を目的とするものである。

【構成】 本発明による合成シリカ粉の製造方法はメチルシリケートをアンモニア水で加水分解、重縮合してコロイダルシリカとしたのち、500 μ m 以下に篩別し、酸化雰囲気下に1,000 ~1,100 $^{\circ}$ Cで加熱処理し、ついで同温度領域においてヘリウムガスを導入して緻密化することを特徴とするものである。

【特許請求の範囲】

【請求項1】メチルシリケートをアンモニア水で加水分解、重縮合してコロイダルシリカとしたのち、500 μm 以下に篩別し、酸化雰囲気下に1,000 ~1,100 $^{\circ}\text{C}$ で加熱処理し、ついで同温度領域においてヘリウムガスを導入して緻密化することを特徴とする合成シリカ粉の製造方法。

【発明の詳細な説明】

【0001】

【産業上の利用分野】本発明は合成シリカ粉の製造方法、特にカサ比重が高く、泡不良のない合成石英ガラスを製造するための合成シリカ原料粉の製造方法に関するものである。

【0002】

【従来の技術】合成石英ガラスが合成シリカ粉の熔融ガラス化により得られることはよく知られているところがあるが、この合成石英ガラスの製造方法についてはベルヌイ法、スート法、ゾルーゲル法、クリストバライトー真空熔融法などが知られている。

【0003】このベルヌイ法は四塩化けい素を酸水素加水分解溶解するものであるが、これにはOH基含有量が1,000ppm程度となるために高温粘度が低いという不利があり、スート法は四塩化けい素を比較的低温の酸水素炎中で加水分解して多孔質ガラス母材を作り、ヘリウムガス雰囲気下、ヘリウムー塩素ガス雰囲気下あるいは真空雰囲気下で加熱溶解し透明ガラス化するものであるが、これもOH基量あるいはCl含有量が300 ppm程度となるので高温粘度が低く、高温で変形するという問題点がある。

【0004】また、このゾルーゲル法はシリケートを加水分解してシリカゾルを作り、これをゲル化して得たゲル化乾燥体を1,200 $^{\circ}\text{C}$ 程度で加熱溶解して透明ガラス化するものであるが、これにはOH基が残り易く、高温粘性の高いものが得られ難く、製造に長時間を要し、さらには製造中にクラックの発生が起きる可能性があるために薄板しかできないという不利があり、このクリストバライトー真空熔融法はフュームドシリカをアルカリ水溶液でゾル化し、凍結乾燥後に焼成してクリストバライト化し、1,700 $^{\circ}\text{C}$ 以上で真空熔融するものであるが、これには得られる石英ガラスが純度のわるいものであり、これが微細な泡をもつものになるという欠点がある。

【0005】

【発明が解決しようとする課題】そのため、本発明者らはこのゾルーゲル法で高温粘性の高い合成石英ガラスを製造する方法について検討を行ない、これについてはシリケートとして特にメチルシリケートを使用し、これをアンモニア水で加水分解、重縮合し、得られたシリカを加熱して未反応の有機物を酸化除去したのち1,500 $^{\circ}\text{C}$ 以上に加熱して真空中で焼結させ、ついで1,700 $^{\circ}\text{C}$ 以上で加熱溶解すれば高温粘性の高い合成石英ガラスを容易

に、かつ安価に得ることができることを見出した（特開平2-1870723号公報参照）が、これにはここに使用される合成シリカ粉がカサ密度の低いもので、無孔性が不充分であると得られる合成石英ガラスが純度が悪く、かつ微泡を含むものになるという不利のあることが判った。

【0006】

【課題を解決するための手段】本発明はこのような不利を解決した合成石英ガラス製造用の合成シリカ粉の製造方法に関するものであり、これはメチルシリケートをアンモニア水で加水分解重縮合してコロイダルシリカとしたのち、500 μm 以下に篩別し、酸化性雰囲気下に1,000 ~1,100 $^{\circ}\text{C}$ で加熱処理し、ついで同温度領域においてヘリウムガスを導入して緻密化することを特徴とするものである。

【0007】すなわち、本発明者らはカサ比重が高く、緻密化された合成シリカ粉の製造方法について種々検討した結果、メチルシリケートをアンモニア水で加水分解し、重縮合させてコロイダルシリカを500 μm 以下に湿式で篩別し、脱水、洗浄後酸化性雰囲気下に1,000 ~1,100 $^{\circ}\text{C}$ で加熱処理したのち、同温度領域にヘリウムガスを導入すると、このシリカ粉がカサ比重が高く、緻密化されたものになるということを見出し、このようにして得たシリカ粉を加熱溶解して合成石英ガラスとすれば純度が高く、泡や異物のない合成石英ガラスを容易に得ることができることを確認して本発明を完成させた。以下にこれをさらに詳述する。

【0008】

【作用】本発明は超高純度で完全無泡の合成石英ガラスを製造するための合成シリカ粉の製造方法に関するものであり、これはメチルシリケートとアンモニア水との加水分解、重縮合で得られたコロイダルシリカを500 μm 以下に湿式で篩別し、脱水、洗浄後酸化性雰囲気中で焼成し、脱水、脱ガスしたのち、1,000 ~1,100 $^{\circ}\text{C}$ でヘリウムガスを導入して緻密化してなることを要旨とするものである。

【0009】本発明による合成シリカ粉の製造はゾルーゲル法で行なわれるが、ここに使用されるシリケートはメチルシリケートとされるし、このメチルシリケートの加水分解はアンモニアの存在で行なわれる。このメチルシリケートをアンモニア水で加水分解し、重縮合させると、この加水分解で生成されるシリカは粒度が500~1,000nmであるシリカの一次粒子となり、この一次粒子は球状で内部には殆んどOH基が含まれず、OH基は表面のみに存在するようになるし、この粒子は開孔気孔径が大きく、温度をかけても開孔化せず、表面のOH基および内部のCは加熱によって容易に除去することができ、さらにはこの三次元重縮合が規則正しく行なわれて構造が密なコロイダルシリカとなる。

【0010】このようにして作られたコロイダルシリカは反応フラスコの壁や攪拌羽根に付着し、それが剥離し

て固いゲル状シリカとなるが、このゲル状シリカは閉孔化が速く、したがって完全に脱水や脱ガスを行うことが難しいので、これは篩別により除く必要があるが、そのために本発明では湿式篩別によりこれは500 μm 以下のものとされる。

【0011】このように篩別されたシリカは酸化性雰囲気での加熱によりここに含有される有機物が除去される（脱炭される）のであるが、このものはその処理に先立って常法によって固液分離することがよくこれは例えば遠心脱水機によって行なえばよい。この遠心脱水ではアンモニアが残存していると、つぎの酸化加熱によってCを充分除去できなくなるので、超純水によってアンモニアやメタノールをよく除去することが必要である。このような固液分離で得られたコロイダルシリカはついで酸化性雰囲気での加熱により脱炭するのであるが、この加熱処理はそれが1,000 $^{\circ}\text{C}$ 未満では脱炭が不充分となるし、1,100 $^{\circ}\text{C}$ より高い温度とすると、凝集粒子の粒子同志の空隙が閉じられて泡となる可能性があるので、これは1,00~1,100 $^{\circ}\text{C}$ で行なう必要があるが、これによれば脱炭と共に脱水も行なわれる。

【0012】本発明はこのようにして脱炭されたシリカ粉をこの処理温度において不活性ガスと接触させるのであるが、この不活性ガスはヘリウムガスとすればよく、これによればシリカ粉の凝集粒子が緻密化されて、ガラスとほぼ同じ密度のものとなり、合成石英ガラス製造時における充填密度を向上させること、また仕込み量を従来より多くすることができるので、品質のすぐれた合成石英ガラスを生産性よく製造することができるし、得られる合成石英ガラスを泡、異物のないものとすることができるという有利性が与えられる。この理由はヘリウムガスを導入すると熱拡散が均一に行なわれ、凝集粒子1ケ1ケの緻密化が凝集粒子同志の焼結より優先して起るためであるが、このような効果はヘリウム以外のガスでは期待できない。

【0013】

【実施例】つぎに本発明の実施例および比較例をあげる。

実施例、比較例

5リットルの連続フラスコにメチルシリケート26.5/時と20.5%のアンモニア水・E Lグレード〔大盛化工

（株）製造商品名〕26.5リットル/時とを25.8リットル/時の滴下速度で同時滴下したところ、10kg/時で凝集コロイダルシリカが得られたのでこれを500 μm の網で篩別した。ついで、このものを一旦放置してからポリプロピレン製1,000#のろ布を設けた遠心脱水機で固液分離をし、超純水で5回洗浄し、得られた凝集コロイダルシリカを石英ガラス炉芯管に詰め、酸素ガス雰囲気下で室温から1,000 $^{\circ}\text{C}$ まで10時間かけて昇温し、2時間保持して脱炭した。

10 【0014】つぎにこの温度を保持したままでこの雰囲気（ヘリウムガス雰囲気）に切りかえて3時間保持したところ、緻密化したシリカ粉が得られたが、この粉体を150~200メッシュとすると共に、これを比較のためにこのヘリウムガス雰囲気下での閉孔化処理を行なわなかったものと比較したところ、タッピング法のカサ比重比較において本発明のものが0.98であるのに対し、比較例のものは0.80であり、このものを2日間放置後に真空加熱してその脱ガス量をしらべたところ、図1に示したように本発明のものがA曲線であるのに対し比較例のものはB曲線の通りであり、本発明品はかなり吸着しにくいものであることが確認された。

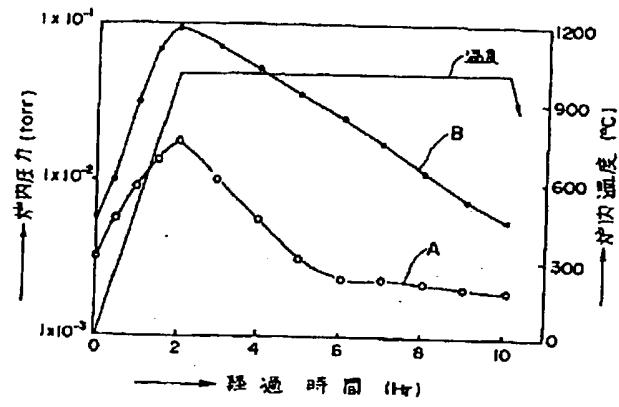
20 【0015】

【発明の効果】本発明は合成シリカ粉の製造方法に関するものであり、これは前記したようにメチルシリケートをアンモニア水で加水分解、重縮合してコロイダルシリカとしたのち、500 μm 以下に篩別し、酸化性雰囲気（ヘリウムガス）に1,000~1,100 $^{\circ}\text{C}$ で加熱処理し、ついで同温度領域においてヘリウムガスを導入して緻密化することを特徴とするものであるが、これによればゾルーゲル法で得られたシリカ粉がカサ比重が高くて無孔化されたものとなるので、これは合成石英ガラス原料とするときに充填密度を向上させること、また仕込み量も多くすることができるので、品質もすぐれた合成石英ガラスを生産性よく製造することができるし、得られる合成石英ガラスを泡不良のないものとすることができるという有利性を与える。

【図面の簡単な説明】

【図1】実施例および比較例で得られた合成シリカ粉の経過時間と脱ガス量との関係グラフを示したものである。

【図1】





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TITLE: Synthetic silica glass powder for producing
quartz glass crucible, is obtained by heating amorphous
silica powder produced by sol-gel process, at preset
temperature in hydrogen atmosphere

PRIORITY-DATA: 2000JP-0076101 (March 17, 2000)

PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE
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BASIC-ABSTRACT:

NOVELTY - The synthetic silica glass powder is obtained by heating an amorphous silica powder produced by sol-gel process, up to 800-1300 deg. C in hydrogen atmosphere.

USE - For producing quartz glass crucible for drawing up silicon single crystal.

ADVANTAGE - The quartz glass crucible suppresses foam generation in its inner layer when heated at high temperature. The silicon single crystal having high purity and improved crystallization rate, is obtained.